



TITLE:

Preparation of Single Crystals of High Melting Point Metals and Alloys Oriented in Any Crystallographic Direction : Production of Molybdenum Resistance Vacuum Furnace

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and (Method 6)

$$\left. \begin{aligned} P &\equiv (k_2 - k_1)\Gamma_2, & Q &\equiv (1 - \Gamma_1\Gamma_2) \\ R &\equiv (k_2 - k_1)\Gamma_1, & S &\equiv k_1k_2(\Gamma_1\Gamma_2 - 1) \end{aligned} \right\} \quad (4)$$

with  $\theta_i \equiv \tan \frac{2\pi}{\lambda_g} x_{0i}$  and  $k_i \equiv \tan \frac{2\pi}{\lambda_g} l_i$ .

In the above experiments, we have ascertained that especially Method 6 is still most practical as mentioned previously and moreover General Method is expressed in a pretty complicated form but is characterized by its merit that  $l_i$  can be selected so that little errors as possible attend the measurement because  $l_1$  and  $l_2$  are arbitrary.

As for the vicinity of large  $|k_i|$  in which large errors accompany the measurement, Method 7 out of methods 1-7 is most convenient to avoid it.

### 3. Preparation of Single Crystals of High Melting Point Metals and Alloys Oriented in Any Crystallographic Direction

#### Production of Molybdenum Resistance Vacuum Furnace

Hideo TAKAKI and Masashige KOYAMA

(H. Takaki Laboratory)

One of the authors has previously succeeded in preparing single crystals oriented in any crystallographic direction with nickel and low silicon steel by the modification of the Bridgman method, using the vacuum Tammann furnace. In that case, the charging of the poly-crystals grown from the single crystal seed was smoothly performed in the shape of wire or ribbon, owing to the plasticity of these materials. However, in the brittle materials (e. g. silicon steel containing the silicon more than five percent), the charging is difficult, as the brittle materials cannot be deformed. The vacuum of the used furnace was also not so high.

Therefore, under the plan of preparing single crystals of brittle materials oriented in any crystallographic direction, by means of charging the brittle materials in the state of small blocks into the upper part of the porcelain tube shown in Fig. 1 and performing both the melting of charges and the preparing of single crystals at the same time, a molybdenum resistance vacuum furnace was produced in order to obtain an equilibrium high temperature region over a long distance and also to obtain a higher vacuum than before.

The vacuum furnace diagrammed in Fig. 2 consists essentially of a carbon steel tube 1 of about 250 mm in diameter and about 1130 mm long, with a resistance wound core 2 of about 600 mm long mounted coaxially in it. The core material is a high purity alundum grooved to accomodate the 1.2 mm molybdenum wire winding. The entire assembly is evacuated from the outlet 9 by means of a two-steps oil diffusion pump, backed by a two-steps rotary pump.

- (1) Steel Tube
- (2) Alundum Tube
- (3) Porcelain Tube
- (4) Molybdenum Wire
- (5) Kanthal Wire
- (6) Glass Window
- (7) Cooler
- (8) Rubber Packing
- (9) Outlet
- (10) Lowering Apparatus
- (11) Reflection Shield
- (12) Support
- (13) Electrode

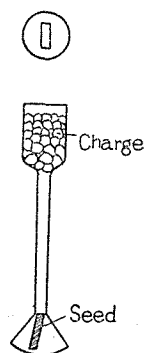


Fig. 1. Tube design.

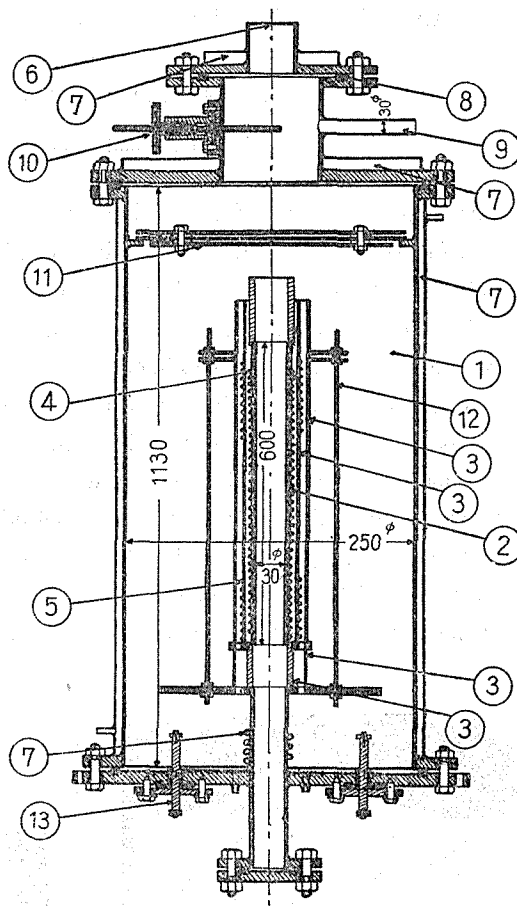


Fig. 2. Furnace diagram.

The space between the alundum tube 2 and the porcelain tube 3 was compacted with  $\alpha$ -alumina powder previously heated at about 1500°C.

The vacuum obtained with the pumping system described was about  $10^{-2} \sim 10^{-3}$  mm of Hg in the molten state of the four percent silicon steel at 1540°C, and the temperature gradient at 1450°C (melting point of four percent silicon steel) was about

40°C/cm.

About one thousand watts of power was sufficient to obtain and maintain 1540°C.

#### 4. X-Ray Studies on the Cast Structure of High Purity Aluminium in the Light of Anisotropy of the Rate of Crystal Growth

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It was assumed by one of the authors that the [001] direction would be the direction of far easier growth than other directions in the case of preparing of single crystals of nickel and low silicon steel, oriented in any crystallographic direction. In the light of this anisotropy of the rate of crystal growth, an X-ray examination was previously performed with the specimens cut off from one part of the ingot of four percent silicon steel (930 kg in weight) produced at the Kawasaki Iron Mfg. Co. However, as the used specimen contained many impurities and gases and its cast structure was very complex, the analysis was very difficult. So in this investigation, the high purity aluminium (99.993 %) was melted and cast in vacuum and a method in which the bottom of the stainless steel mould was simultaneously water-cooled after casting the molten metal in it, was devised so as the columnar crystals were developed perpendicularly to the bottom of ingot.

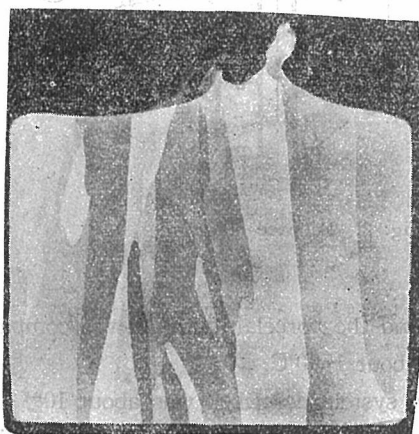


Fig. 1, Longitudinal section.



Fig. 2, Transversal section.